Patent-Treuhand-Gesellschaft für elektrische Glühlampen mbH

Process and apparatus for producing oxidic nanocrystals

CLAIMS:

- A process for producing oxidic nanocrystals, 5 which comprises the following steps:
 - a) Introducing host lattice ions as metalorganic complexes or volatile inorganic compounds into at least a first vaporizer (12),
- b) Converting the complexes or compounds into the 10 gas phase and
 - c) Transporting the resultant gas phase, with the aid of one or more carrier gases and/or carrier gas mixtures, into at least one heating zone (18)of at least one reactor (16), supplying one or more reaction gases and/or reaction gas mixtures,
 - d) Breaking down the complexes or compounds in the heating zone (18) of the reactor (16) at a pressure of between 1 1000 mbar, immediately forming the oxidic nanocrystals, and
 - e) Collecting the oxidic nanocrystals at at least one adsorption trap (20).
 - The process as claimed in claim 1, wherein in process step a), together with the host lattice ions, doping ions are introduced into a second vaporizer (14) metalorganic complexes or as volatile inorganic compounds, and the complexes or compounds are converted into the gas phase, and the resultant gas phase is transported into the heating zone (18) of the reactor (16) with the aid of one or more carrier gases and/or carrier gas mixtures.
 - The process as claimed in one of the preceding claims, wherein the host lattice ions are elements from main groups II to VI of the Periodic System of the

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Elements, in particular rare-earth elements, preferably Y, Gd, and Mg, Ca, Ba and Al.

- 4. The process as claimed in one of the preceding 5 claims, wherein the doping ions are ions of the rare-earth elements.
- 5. The process as claimed in one of the preceding claims, wherein the metalorganic complexes are compounds of the host lattice ions or doping ions with hydrogen tetramethylheptanedionate.
- 6. The process as claimed in one of claims 2 to 5, wherein the resultant doped, oxidic nanocrystals are phosphors.
 - 7. The process as claimed in one of the preceding claims, wherein the first and second vaporizers (12, 14) are at a temperature of approximately 30-900°C.
 - 8. The process as claimed in one of the preceding claims, wherein the temperature in the heating zone (18) of the reactor (16) is approximately 600-1600°C.

- 25 9. The process as claimed in one of the preceding claims, wherein the carrier gases used are inert gases or gas mixtures.
- 10. The process as claimed in one of the preceding 30 claims, wherein the reaction gas used is oxygen or mixtures thereof.
- 11. The process as claimed in one of the preceding claims, wherein the flow rate of the carrier gas or the 35 carrier gas mixtures in the system is 20 ml/min-50 l/min.
 - 12. The process as claimed in one of the preceding claims, wherein the flow rate of the reaction gas or

the reaction gas mixtures in the system is 50 ml/min-100 l/min.

- 13. The process as claimed in one of the preceding claims, wherein the pressure in the entire system is approx. 1-1500 mbar.
- 14. The process as claimed in one of the preceding claims, wherein the resultant oxidic nanocrystals are present in the thermodynamic equilibrium phase.
 - 15. The process as claimed in claim 14, wherein the resultant oxidic nanocrystals have an initial particle size of at most 100 nm, preferably at most 20 nm, and a cubic crystal structure.

- 16. An apparatus for producing oxidic nanocrystals, which comprises at least one vaporizer (12, 14), at least one reactor (16) with at least one heating zone (18), at least one adsorption trap (20, 22) and at least one vacuum pump (24).
- 17. The apparatus as claimed in claim 16, wherein the vaporizers (12, 14) have vaporizer heating means which are designed as oil baths.
 - 18. The apparatus as claimed in claim 16 or 17, wherein the reactor (16) is a flow reactor.
- 30 19. The apparatus as claimed in one of claims 16 to 18, wherein in each case one heatable tube (48, 50) is arranged between the vaporizer(s) (12, 14) and the reactor (16).
- 35 20. The apparatus as claimed in one of claims 16 to 19, wherein the reactor (16) comprises an aluminum tube (52) and a cylindrically designed heating zone (18).

- 21. The apparatus as claimed in claim 20, wherein a baffle (40) is arranged inside the reactor (16) at the end of the heating zone (18).
- The apparatus as claimed in one of claims 16 to 21, wherein a filter is arranged between the second adsorption trap (22), which is connected downstream of the first adsorption trap (20), and the vacuum pump (24).

- 23. The apparatus as claimed in one of claims 16 to 22, wherein the adsorption traps (20, 22) are cold traps.
- 15 24. The use of the oxidic nanocrystals produced using the process as claimed in one of claims 1 to 15 as phosphors for illumination equipment with low or high luminances.
- 25. A phosphor for illumination equipment, having a host lattice comprising oxidic crystals, in particular oxides or oxysulfides, of the metals Y, Gd, Mg, Ca, Ba, Al, in which the host lattice is activated by rare-earth elements, the phosphor comprising nanocrystals with a low level of agglomeration with a
- nanocrystals with a low level of agglomeration, with a mean ultimate particle size of 1 to 20 nm and a cubic crystal structure.
- 26. The phosphor as claimed in claim 25, wherein the absorption edge of the host lattice is shifted toward shorter wavelengths compared to a standard of the same material.